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IS 1448-141 (1992): Methods of test for petroleum and its products, Part 141: Determination of apparent density of petroleum coke by helium method [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS 1448 [P : 141] : 1992

(Reaffirmed 2003)

भारतीय मानक

पेट्रोलियम और इसके उत्पादों की परीक्षण पद्धतियां

[पी : 141]

हेलियम पद्धति द्वारा पेट्रोलियम कोक का प्रत्यक्ष घनत्व ज्ञात करना

Indian Standard

METHODS OF TEST FOR PETROLEUM
AND ITS PRODUCTS

[P : 141]

DETERMINATION OF APPARENT DENSITY OF PETROLEUM
COKE BY HELIUM METHOD

UDC 665.777.4 : 531.756

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Price Group 2

Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 1

FOREWORD

This Indian Standard [P : 141] was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

*Indian Standard***METHODS OF TEST FOR PETROLEUM
AND ITS PRODUCTS**

[P : 141]

**DETERMINATION OF APPARENT DENSITY OF PETROLEUM
COKE BY HELIUM METHOD****1 SCOPE**

This method covers the determination of the volume of a sample of petroleum coke from which the helium apparent density is calculated.

2 SIGNIFICANCE AND USE

2.1 The process of making anodes of as high a percentage of carbon as possible requires certain parameters to be of designated specifications. One such parameter is the helium apparent density. The knowledge of the helium apparent density in combination with the mercury apparent density allows the calculation of the open porosity of the coke.

2.2 Open porosity may be determined knowing the mercury apparent density and helium density of coke and thus the amount of pitch to be added for anode production.

3 OUTLINE OF THE METHOD

Beckman apparatus consists of two chambers and two pistons as shown in Fig. 1. For the purpose of illustration the chambers are assumed to be equal in volume and there is no sample in either cylinder. With the connecting valve closed, any change in the position of one piston shall be duplicated by an identical stroke in the other in order to maintain the same pressure on each side of the differential pressure indicator. If the connecting valve is closed and both pistons are advanced the same amount to position 2 with the sample inserted in chamber B, the pressure will not remain the same. However, the pressure in the two chambers may be made equal by withdrawing piston B from position 2 to position 3, an amount equivalent in volume to v_x . If piston A, always is advanced exactly the same distance each time a measurement is made, the distance that piston B differs from piston 2, when the pressure in both cylinders is equal will always be proportional to the volume v_x .

The distance between 2 and 3 (d_x) is calibrated and made to read directly in terms of cm^3 . A digital counter is employed to indicate the distance d_x , so that the instrument can be read directly in cm^3 , knowing the volume and mass of the sample, the density is calculated.

4 REAGENTS

Helium gas, 99.9 percent purity.

5 APPARATUS

5.1 Air comparison Pyknometer with external purge manifold attachment (see Fig. 2 for attachment).

NOTE — The test procedure described here is specific to Beckman Air Comparison Pyknometer model 930 from Beckman Instruments Inc. Fullerton, CA 92634, USA. Similar instruments by which identical results may be obtained are also suitable.

5.2 Sample Cup

5.3 Test Ball, 8.58 ml.

5.4 Test Ball, 28.96 ml.

5.5 Rotary High Vacuum Pump, capable of producing a vacuum up to 0.001 mm of mercury.

5.6 Vacuastat, capable of measuring vacuum up to 0.001 mm of mercury.

5.7 Analytical Balance

5.8 Drying Oven, maintained at 105-110°C.

5.9 Jaw Crusher, laboratory type, manganese steel jaw plates.

5.10 Sieves, Tyler No. 10 (mesh aperture 1.65 mm) or equivalent.

Tyler No. 20 (mesh aperture 0.85 mm) or equivalent.

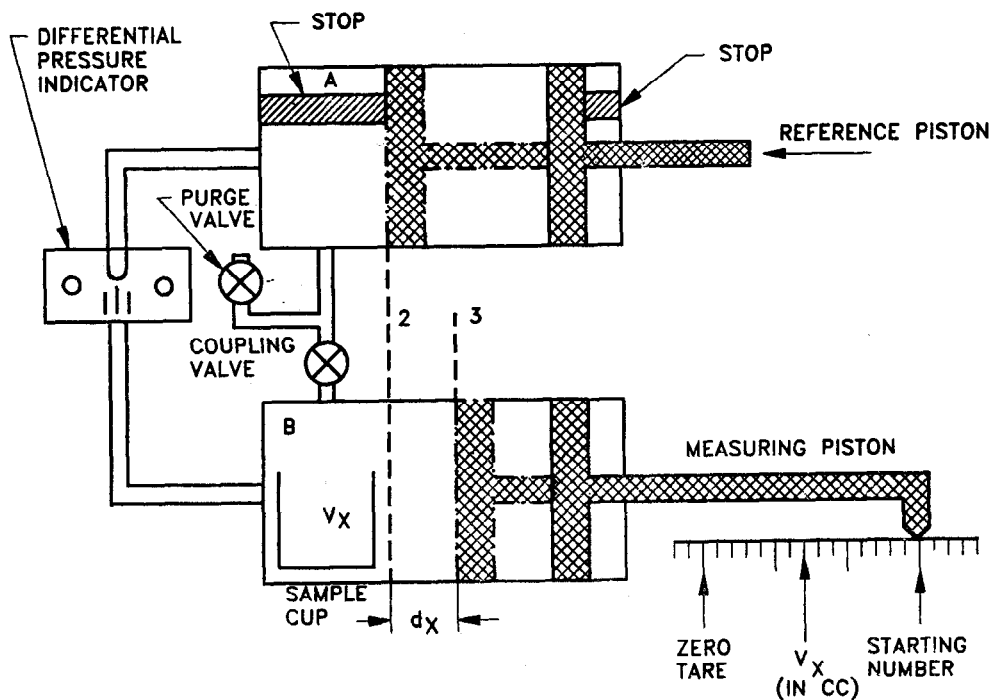


FIG. 1 SIMPLIFIED SCHEMATIC DIAGRAM

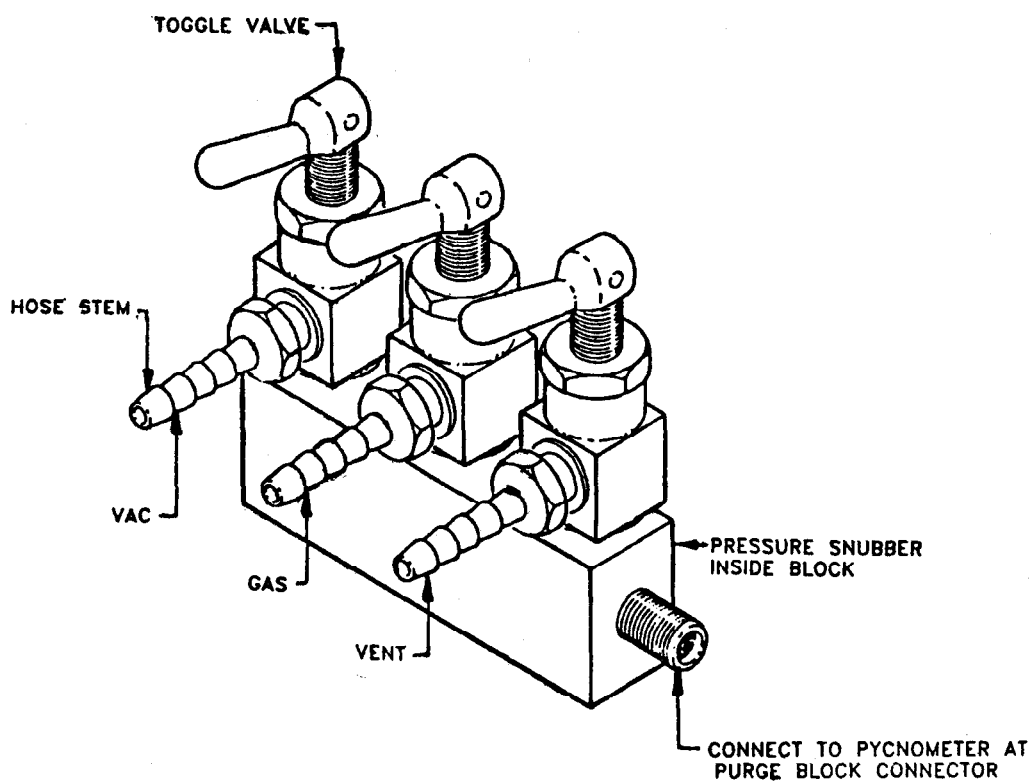


FIG. 2 EXTERNAL PURGE MANIFOLD ATTACHMENT (ACCESSORY)

6 SAFETY PRECAUTIONS

Whenever dealing with a vacuum, safety glasses shall be worn.

7 SAMPLE PREPARATION

7.1 The representative sample is passed through a Jaw crusher rated to give grains smaller than 1.65 mm.

7.2 The crushed coke is shifted and the —1.65 mm to + 0.85 mm fraction is kept for analysis.

7.3 The fraction is dried for one hour in an oven maintained at 105-110°C and then cooled in a desiccator.

8 PROCEDURE

8.1 Purge 1 to 2 atmosphere operation.

8.2 Before beginning sample measurements, make a zero measurement check, as described in **8.3** to compensate, if necessary for possible zero offset. Then proceed as follows.

8.2.1 The dried sample (10 g) is added to weighed sample cup.

8.2.2 Connect external valving attachment. Connect gas valve to the inert gas supply and vacuum valve to the vacuum line. Set the gas inlet pressure for about 2 psig (13.8 KPa).

8.2.3 Rotate both handwheels to counter clockwise extreme.

8.2.4 Turn measuring handwheel clockwise until starting number is set on the counter, starting number is stamped on case above measuring handwheel.

8.2.5 Insert cup with sample in compartment. Lock sample cup in place by pressing handle down firmly.

8.2.6 Open purge valve. Open coupling valve.

8.2.7 Open vacuum valve and allow system to evacuate to the desired pressure (0.1 mm of Hg). Close vacuum valve.

8.2.8 Open gas valve and allow about 5 seconds for pressure equilibration. Close gas valve.

8.2.9 Open vent valve and allow about 5 seconds for pressure equilibration. Close vent valve and the purge valve.

8.2.10 Wait 10 seconds, then close coupling valve.

8.2.11 Turn both handwheel clockwise simultaneously or alternately until reference handwheel rest against stop. Keep pointer on scale during this process.

8.2.12 Wait 10 seconds. Bring pointer to null with measuring wheel.

8.2.13 Open coupling valve. Read sample volume on counter directly in ml.

8.2.14 Turn both handwheels counter clockwise to rest against stops. Remove sample cup and weigh.

Mass of sample = wt (**8.2.14**) — wt (**8.2.1**)

8.2.15 When maximum precision measurements are required, the time allotted for temperature equilibration should be at least 3 to 5 times longer.

NOTE — When opening coupling valve, observe null indicator. If it shifts position, true null was not obtained and the run should be repeated.

8.3 Zero Measurement Check

8.3.1 The zero check procedure is identical to the measurement procedure, but is made with the sample cup clean and empty. If the final counter reading is other than zero, repeat the zero measurement check to verify the zero offset.

8.3.2 To correct the zero offset, use the following procedure:

- i) If the zero counter reading is greater than zero, use it as a tare number;
- ii) Subtract from the subsequent sample volume measurements; and
- iii) If the zero counter reading is less than zero, add the difference between the reading and zero to subsequent sample measurement.

8.4 Calibration Check

Accuracy of the unit can be checked by making measurements with the two test balls supplied. Measurements procedure is identical to that already given, except that the test ball is measured in the sample cup in place of sample. Take several measurements with each test ball. The 38.1 mm calibration ball has a volume of 28.96 ± 0.015 ml. The 25.4 mm ball volume is $8.58 \text{ ml} \pm 0.015 \text{ ml}$.

8.5 All experimental procedures shall be carried out at $25 \pm 1^{\circ}\text{C}$.

9 CALCULATION

The result appearing on the Pyknometer is the volume of the sample. Density is obtained by dividing the mass of sample by the volume.

10 PRECISION

Results of duplicate tests shall not differ by more than the following amounts:

<i>Repeatability</i>	<i>Reproducibility</i>
0.01 from the mean result	0.02 from the mean result

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